



**APPENDIX D. ANALYTICAL PROTOCOLS,
TECHNICAL BACKGROUND DOCUMENT: MERCURY WASTES,
EVALUATION OF TREATMENT OF BULK ELEMENTAL MERCURY,
FINAL REPORT**

FEBRUARY 8, 2002

**SUBMITTED FOR:
U.S. ENVIRONMENTAL PROTECTION AGENCY
ARIEL RIOS BUILDING
OFFICE OF SOLID WASTE
1200 PENNSYLVANIA AVENUE, N.W.
WASHINGTON, D.C. 20460**

**SUBMITTED BY:
SCIENCE APPLICATIONS INTERNATIONAL CORPORATION
ENGINEERING AND ENVIRONMENTAL MANAGEMENT CORP.
11251 ROGER BACON DRIVE
RESTON, VIRGINIA 20190**

**EPA CONTRACT No. 68-W-98-025
WORK ASSIGNMENT No. 3-8
SAIC PROJECT No. 06-0758-08-1373-000**



APPENDIX D. ANALYTICAL PROTOCOLS

SOP Titles

Procedure for the Determination of Ammonium Acetate Extractable Calcium, Magnesium,
Sodium and Potassium in SoilD-3

Undisturbed Bulk Density Analytical ProcedureD-10

Determining the Cation Exchange Capacity (CEC) of SoilD-12

pH Analytical ProcedureD-14

Determining the Acidity of SoilD-17

Hydraulic Conductivity — Constant Head MethodD-20

Texture Procedure — Pipette MethodD-23

Redox Potential of a Soil SampleD-28

Cation-Exchange Capacity by Sodium Saturation in Calcareous SoilsD-31

Determination of Total Organic Carbon in Soil by CombustionD-35

1/3 Bar — German MethodD-38

ChloridesD-39





This page intentionally left blank.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.12.13

SOP TITLE: Procedure for the Determination of Ammonium Acetate Extractable Calcium, Magnesium, Sodium and Potassium in Soil

PROCEDURE:

1. To extract the samples, use a 1.0 gm soil scoop to transfer the sample to 50 ml Erlenmeyer flasks in Potassium extraction tray, according to SOP NUT.06.02 – “Soil Scoop Procedure.” Scoop a check sample after the last sample in each tray.
2. Use an appropriate dispenser or a dispenser pump to add 10.0 ml of Potassium Extracting Solution to each sample. For each batch of samples, check one aliquot with a graduated cylinder to determine the actual volume dispensed and record the volume on the raw data lab sheet. This volume is to be at 10.0 ml or reject and recalibrate and check volume again.
3. Shake for 5 minutes at 200 ($\pm 10\%$) rpm on a mechanical shaker.
4. Filter through sodium-free 9 cm filter paper into an ICP filtering tray or into plastic cups in Potassium filter trays, labeled the same as the extracting tray.
5. Remove the filter papers and read the extract on the ICP according to SOP NUT.01.25 – “Optima 3000 & 3100 Operating, Calibration and Maintenance Procedure” and SOP NUT.02.93 – “Determination of Ammonium Acetate Extractable Calcium, Magnesium, Sodium, and Potassium for Cation Exchange Capacity using ICP”. Record the results on the raw data lab sheet or capture electronically. For all samples: Read the standards after every tray. If the read standard is more than $\pm 10\%$ of the known standard, the run must be re-tested. Record the standard readings on the raw data lab sheet. The check sample must fall within the limits set in SOP NUT.04.03 – “The Use and Ranges of Check Soil”.
6. Samples may also be analyzed using the Atomic Absorption Spectrophotometer. Use Steps 6 through 10 when using the Atomic Absorption Spectrophotometers. Read the extracts for Potassium and Sodium on the Atomic Absorption Spectrophotometer, according to SOP NUT.01.21 – “Atomic Absorption Spectrophotometer Operating Procedure – Buck Scientific Models” & NUT.01.23 – “Atomic Absorption Spectrophotometer Operating Procedure – Perkin-Elmer 100”.
7. **For Calcium and Magnesium:**
 - A) To prepare samples, use either step a), b), or c).
 - a) Use a 1000 μ l auto-pipet to take a 1.00 ml aliquot of each sample, transferring them to cups in a CEC tray, numbered the same as the Potassium tray. Use a pipet or dispenser to add 1.00 ml of 2% Lanthanum Chloride Solution. Use a dispenser to add 8.00 ml of deionized water.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.12.13

SOP TITLE: Procedure for the Determination of Ammonium Acetate Extractable Calcium, Magnesium, Sodium and Potassium in Soil

- b) Use the Hamilton Microlab Dilutor to dispense 1000 µl of sample and 9000 µl of the 1:9 Dilution of 2% Lanthanum Chloride Solution.
 - c) Use a 1000 µl auto-pipet to take a 1.00 ml aliquot of each sample, transferring them to cups in a CEC tray, numbered the same as the Potassium tray. Use a dispenser to add 9.00 ml of the 1:9 Dilution of 2% Lanthanum Chloride Solution.
- B) Read on the Atomic Absorption Spectrophotometer, according to SOP NUT.01.21 – “Atomic Absorption Spectrophotometer Operating Procedure – Buck Scientific Models” & NUT.01.23 – “Atomic Absorption Spectrophotometer Operating Procedure – Perkin-Elmer 100”.
- C) Record the results on the raw data lab sheet or capture electronically.
- a) If the samples are low in calcium and/or magnesium and levels can not be detected, the following procedure may be used:
 - 1) Use a 1 gm soil scoop and transfer soil two times for a total of 2 gm of soil to an Erlenmeyer flask in a potassium extraction tray. Use an appropriate dispenser and add 20 ml of Potassium Extracting Solution to each sample. Shake for 5 minutes on a mechanical shaker. Filter through 11 cm filter paper into plastic cups. This extracting method is needed in order to have at least 10 ml of extract.
 - 2) Use a pipet to transfer 9 ml of each sample to a plastic cup. Use a pipet or dispenser to add 1.0 ml of 2% Lanthanum Chloride to each sample.
 - 3) Read on the AA and calculate by:
$$\text{Sample (ppm)} = \text{AA Reading}/9$$
Record the calculated reading on the raw data lab sheet.
- D) Check all pipets and dispensers with a graduated cylinder to determine actual volume dispensed and record the volume on the raw data lab sheet. This should be at exact volumes, dispensed or reject and calibrate pipets and check volumes again.
8. For all samples: Read the standards after every tray. If the read standard is more than ±10% of the known standard, the run must be re-tested. Record the standard readings on the raw data lab sheet.
9. If the sample exceeds the highest standard by more than 10%, an appropriate dilution is made.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.12.13

SOP TITLE: Procedure for the Determination of Ammonium Acetate Extractable Calcium, Magnesium, Sodium and Potassium in Soil

10. The check sample must fall within the limits set in SOP NUT.04.03 – “The Use and Ranges of Check Soil”. If the check soil does not fall within these limits, the lab manager or technician III may approve the check soil(s) or may have the samples re-tested.

REAGENTS:

- **POTASSIUM EXTRACTING SOLUTION**

(1N Ammonium Acetate):

	<u>10 L</u>	<u>50 L</u>
Ammonium Hydroxide	690 ml	3450 ml
Acetic Acid	600 ml	3000 ml

1. Rinse out the carboy with deionized water then fill about 3/4 full.
2. IN (OR IN FRONT OF) THE FUME HOOD use a graduated cylinder to add the Ammonium Hydroxide. Rinse the cylinder with Deionized Water into the carboy.
3. Use the graduated cylinder to add the Acetic Acid. Rinse the cylinder with Deionized Water into the carboy.

DANGER: Ammonium Hydroxide and Acetic Acid both are corrosive and produce corrosive fumes. Use gloves and face protection when handling. Work in (or in front of) the fume hood to avoid inhaling the fumes.

4. Mix the solution by shaking with the lid on, with an electric mixer, or a magnetic stir plate and bar.
5. Fill the carboy to volume with deionized water and mix again.
6. Check the pH and adjust to 8.5. If the pH is over 8.5, add Acetic Acid. If under 8.5, add Ammonium Hydroxide.

(1.0 N Ammonium Acetate Concentrate):

1. Add 400 ml of concentrate to a 2000 ml volumetric flask.
2. Dilute to volume. Mix the solution thoroughly.
3. Adjust the pH of this solution to 8.5.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.12.13

SOP TITLE: Procedure for the Determination of Ammonium Acetate Extractable Calcium, Magnesium, Sodium and Potassium in Soil

- **2% LANTHANUM CHLORIDE SOLUTION:** 2000 ml

Lanthanum Oxide	47.00 g
Cesium Chloride	12.60 g
Hydrochloric Acid	72.0 ml

1. Weigh the Lanthanum Oxide and transfer it to a flask.
2. Weigh the Cesium Chloride and add it to the flask.
3. Fill about 2/3 full with Deionized Water and swirl to mix.
4. Use a graduated cylinder to measure the Hydrochloric Acid and add it to the flask.
5. Swirl until dissolved.
6. Bring to volume with deionized water and mix thoroughly.

- **1:9 DILUTION OF 2 % LANTHANUM CHLORIDE SOLUTION:**
Dilute 250 ml of 2 % Lanthanum Chloride solution to 2000 ml with deionized water in a volumetric flask.

STANDARDS: FOR THE ICP:

- **ICP High CEC Std. – (Calcium 1500 ppm, Magnesium 300 ppm, Sodium 40 ppm, and Potassium 72 ppm in solution) (Calcium 15,000 ppm, Magnesium 3000 ppm, Sodium 400 ppm, and Potassium 720 ppm in soil)**
 1. 80 mls of 1000 ppm Sodium Standard.
 2. 144 mls of 1000 ppm Potassium Standard.
 3. 7.492gms of oven dried Calcium Carbonate.
 4. 0.995 gms of oven dried Magnesium Oxide.
 5. 20 mls of concentrated Hydrochloric Acid.
 6. Dilute to 2000 mls with Potassium Extraction Solution.
- **ICP Medium CEC Std. – (Calcium 1000 ppm, Magnesium 200 ppm, Sodium 20 ppm, and Potassium 36 ppm in solution) (Calcium 10,000 ppm, Magnesium 2000 ppm, Sodium 200 ppm, and Potassium 360 ppm in soil)**
 1. 40 mls of 1000 ppm Sodium Standard.
 2. 72 mls of 1000 ppm Potassium Standard.
 3. 4.995gms of oven dried Calcium Carbonate.
 4. 0.663 gms of oven dried Magnesium Oxide.
 5. 20 mls of concentrated Hydrochloric Acid.
 6. Dilute to 2000 mls with Potassium Extraction Solution.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.12.13

SOP TITLE: Procedure for the Determination of Ammonium Acetate Extractable Calcium, Magnesium, Sodium and Potassium in Soil

- **ICP Low CEC Std. – (Calcium 500 ppm, Magnesium 100 ppm, Sodium 10 ppm, and Potassium 16 ppm in solution) (Calcium 5000 ppm, Magnesium 1000 ppm, Sodium 100 ppm, and Potassium 160 ppm in soil)**
 1. 20 mls of 1000 ppm Sodium Standard.
 2. 32 mls of 1000 ppm Potassium Standard.
 3. 2.497gms of oven dried Calcium Carbonate.
 4. 0.332 gms of oven dried Magnesium Oxide.
 5. 20 mls of concentrated Hydrochloric Acid.
 6. Dilute to 2000 mls with Potassium Extraction Solution.

FOR THE ATOMIC ABSORPTION SPECTROPHOTOMETER:

- **Potassium and Sodium:**
 1. Pipet 1000 ppm Potassium and Sodium Standards into 500 ml volumetric flasks, according to the chart below.
 2. Dilute to volume with Potassium Extracting Solution, and mix thoroughly.

	<u>STANDARD #1</u>			<u>STANDARD #2</u>		
	<u>ml</u>	<u>ppm</u>	<u>ppm in soil</u>	<u>ml</u>	<u>ppm</u>	<u>ppm in soil</u>
Potassium	8	16	160	18	36	360
Sodium	5	10	100	10	20	200

	<u>STANDARD #3</u>		
	<u>ml</u>	<u>ppm</u>	<u>ppm in soil</u>
Potassium	36	72	720
Sodium	20	40	400

- **Calcium and Magnesium:**
 1. Pipet 1000 ppm Calcium and Magnesium Standards into 500 ml volumetric flasks, according to the chart below.
 2. Add 50 ml Lanthanum Chloride Solution.
 3. Add 50 ml Extracting Solution.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.12.13

SOP TITLE: Procedure for the Determination of Ammonium Acetate Extractable Calcium, Magnesium, Sodium and Potassium in Soil

4. Dilute to volume with Deionized Water and mix thoroughly.

		<u>STANDARD #1</u>			<u>STANDARD #2</u>	
	<u>ml</u>	<u>ppm</u>	<u>ppm in soil</u>	<u>ml</u>	<u>ppm</u>	<u>ppm in soil</u>
Calcium	25	50	50	50	100	100
Magnesium	5	10	100	10	20	200

		<u>STANDARD #3</u>	
	<u>ml</u>	<u>ppm</u>	<u>ppm in soil</u>
Calcium	75	150	150
Magnesium	15	30	300

REFERENCES:

Brown, J.R., and Darryl Warncke. 1988. Recommended Cation Tests and Measures of Cation Exchange Capacity. *In* W.C. Dahnke, (ed.). Recommended Chemical Soil Test Procedures for the North Central Region. North Central Region Publication No. 221 (Revised). North Dakota Agricultural Experiment Station Bulletin 499 (Revised).



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.02.05

SOP TITLE: Undisturbed Bulk Density Analytical Procedure

PURPOSE: To define the analytical method for determining the bulk density of undisturbed soil samples received in sampling tubes.

PROCEDURE:

1. Determine the diameter of the soil tube. Use the inside diameter of the soil probe used to take the sample if it is known. Otherwise use a ruler or caliper to measure the inside diameter of the soil tube itself. Record the value to the nearest 0.1cm.
2. Measure the length of the soil core and record the total length to the nearest 0.1cm. If multiple cores represent the sample, sum the lengths of the individual cores that make up the sample.
3. Calibrate the balance following SOP NUT.01.01 – “Use and Maintenance of Laboratory Balances.”
4. Weigh a tray (or several trays) suitable to hold all the soil from the core, and record the weight(s) to at least the nearest 0.1g.
5. Place all the soil on the tray(s) and dry in restricted access soil dryer at $105 \pm 5^{\circ}\text{C}$ for at least 12 hours.
6. Weigh the tray(s) containing the dry soil and record the weight(s) to at least the nearest 0.1g.
7. Calculate the bulk density:

$$\text{Bulk Density} = \frac{\text{Dry Soil Weight}}{\text{Pi} * (\text{Core Radius})^2 * (\text{Core Length})}$$

REFERENCE:

Blake, G.R. 1965. Bulk Density. *In* Methods of Soil Analysis, Part 1. American Society of Agronomy. Madison, WI.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.02.03

SOP TITLE: Determining the Cation Exchange Capacity (CEC) of Soil

PURPOSE: Determining the Cation Exchange Capacity (CEC) of a soil sample.

PROCEDURE:

1. Determine the calcium, magnesium, sodium and potassium of the soil sample by following the procedures described in SOP NUT.02.12 – “Procedures for the Determination of Ammonium Acetate Extractable Calcium, Magnesium, Sodium and Potassium in Soil”.
2. Determine the acidity of the soil sample by following the procedures described in SOP NUT.02.11 – “Determining the Acidity of Soil”.
3. Calculate the CEC using one of the following methods:

Method 1:

lbs per acre Ca / 400 = Meq per 100 gm Ca
lbs per acre Mg / 240 = Meq per 100 gm Mg
lbs per acre K / 780 = Meq per 100 gm K
lbs per acre Na / 460 = Meq per 100 gm Na
 $8 \times (8.00 - \text{buffer pH}) = \text{Meq per 100 gm H}$

CEC = sum of the Meq per 100 gm (Ca + Mg + K + Na + H)

Method 2:

ppm Ca / 200 = Meq per 100 gm Ca
ppm Mg / 120 = Meq per 100 gm Mg
ppm K / 390 = Meq per 100 gm K
ppm Na / 230 = Meq per 100 gm Na
 $8 \times (8.00 - \text{buffer pH}) = \text{Meq per 100 gm H}$

CEC = sum of the Meq per 100 gm (Ca + Mg + K + Na + H)



**AGVISE Laboratories, Inc.
NORTHWOOD, ND**

STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.05.08

SOP TITLE: pH Analytical Procedure

REVISED BY: Linda M. Anderson

TITLE: Laboratory Technician III

REPLACES SOP NUMBER: NUT.02.05.07

REASON FOR REVISION: Update Title Page and Minor Revisions.

APPROVALS:

Robert Deutsch Date
President/Nutrient Laboratory Manager

Julie M. Johnson Date
Quality Assurance Manager

Robert Wallace Date
CEO

Effective Date _____



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.05.08

SOP TITLE: pH Analytical Procedure

PURPOSE: To define the analytical method used to determine soil pH.

EQUIPMENT:

- pH Meter
- Soil Scoop

PROCEDURE:

1. Set up pH/Salt trays, putting small plastic cups in each space.
2. Mark each cup with its appropriate sample number.
3. Using a 5 gm soil scoop, scoop the soil samples into the cups, according to SOP NUT.06.02 – “Soil Scoop Procedure.” Scoop at least one check sample per 10 samples.
4. Use a dispenser to add 5 ml of deionized water to each sample. For each batch of samples, check one aliquots of water with a graduated cylinder to determine the actual volume dispensed, and record the volume on the raw data lab sheet.
5. Stir the samples, and then wait 10 minutes before reading them.
6. Calibrate the pH meter before reading each batch:
 - a. Use the appropriate instruction manual for the pH meter being used and follow the instructions for calibrating the meter.
 - b. Use pH buffers 4.00, 7.00 and 10.00 to calibrate the pH meter.
 - c. Enter the buffers at the end of the run on the raw data lab sheet to see if the meter held the calibration. Acceptable Ranges are as follows:

	<u>4.00</u>	<u>7.00</u>	<u>10.00</u>
Min	3.94	6.94	9.94
Max	4.06	7.06	10.06

7. Fill an adequately large enough beaker with deionized water for rinsing the electrode. Put clean water in the beaker for each run.
8. RINSE THE ELECTRODE IN THE DEIONIZED WATER BEAKER BEFORE EACH SAMPLE.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.05.08

SOP TITLE: pH Analytical Procedure

9. Read each sample by stirring it with the electrode until a fairly steady reading is obtained. THE READING SHOULD BE TAKEN WHILE THE SAMPLE IS BEING STIRRED.
10. Record the reading for each sample on the raw data lab sheet.
11. Turn off the pH meter after all the samples are read and store the electrodes in pH 7 or pH 4 buffer.

REAGENTS:

pH ELECTRODE FILLING SOLUTION (4M KCl): 500 ml
Potassium Chloride 150 g

1. Weigh the Potassium Chloride into a beaker.
2. Add about 400 ml deionized water and stir on a magnetic stirrer until dissolved. Warm slightly if necessary.
3. Dilute to volume with deionized water and stir until mixed.

REFERENCES:

Ecken, D.J. 1988. Recommended pH and Lime Requirement Tests. W.C. Dahnke, (ed.). Recommended Chemical Soil Test Procedures for the North Central Region. North Central Region Publication No. 221 (Revised). North Dakota Agricultural Experiment Station Bulletin 499 (Revised).

Peck, T.R. 1988. Standard Soil Scoop. W.C. Dahnke, (ed.). Recommended Chemical Soil Test Procedures for the North Central Region. North Central Region Publication No. 221 (Revised). North Dakota Agricultural Experiment Station Bulletin 499 (Revised).



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.11.09

SOP TITLE: Determining the Acidity of Soil

PURPOSE: To determine the acidity of a soil sample for use in calculating the CEC of the soil.

SCOPE: Using the Adam-Evans method to determine the Acidity of a soil sample. Ref.: Soil Testing and Plant Analysis Laboratory Manual, 1989, J. Benton Jones, Jr.

EQUIPMENT REQUIRED:

1. pH meter

BUFFER SOLUTION:

1. Using a one-Liter flask with a magnetic stirrer, dissolve 74 g of potassium chloride in approximately 500 ml of water.
2. Add 10.5 g of potassium hydroxide, stir and dissolve.
3. Add 20 g of p-nitrophenol, stir and dissolve.
4. Add 15 g of boric acid.
5. Dilute to 1 L with water and stir. You may need to heat to dissolve.

METHOD:

1. To the 5 grams of soil and 5 ml of deionized water used for pH of soil (Refer to SOP NUT.02.05 – pH Analytical Procedure), add 5 ml of Adams-Evans buffer. Scoop a check sample after the last sample in each tray. Check the dispenser with a graduated cylinder and record this amount on the raw data lab sheet.
2. Shake or stir for 10 minutes.
3. Let stand for 30 minutes.
4. Prepare a standard by mixing 5 ml of deionized water with 5 ml of Adams-Evans buffer solution. Use this solution to calibrate the pH meter to 8.00. The Acceptable Range is as follows:
Min – 7.92
Max – 8.08
5. Read samples and record pH to nearest 0.01 on the raw data lab sheet for Acidity. This reading is the buffer pH.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.11.09

SOP TITLE: Determining the Acidity of Soil

CALCULATIONS:

$$\text{meq acidity (H}^+\text{) /100 g} = 8 \times (8.00\text{-buffer pH})$$



**AGVISE Laboratories, Inc.
NORTHWOOD, ND**

STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.34.03

SOP TITLE: Hydraulic Conductivity — Constant Head Method

REVISED BY: Garis Pollert

TITLE: Nutrient Laboratory Analyst

REPLACES SOP NUMBER: NUT.02.34.02

REASON FOR REVISION: Update Cover Page, Procedures and Minor Revisions

APPROVALS:

Robert Deutsch Date
President/Nutrient Laboratory Manager

Julie M. Johnson Date
Quality Assurance Manager

Robert Wallace Date
CEO

Effective Date _____



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.34.03

SOP TITLE: Hydraulic Conductivity — Constant Head Method

PURPOSE: To define the analytical method for determining the hydraulic conductivity of a saturated soil.

EQUIPMENT:

- Constant-Head Hydraulic Conductivity Apparatus (See Figure 1).
- Container to hold water.

PROCEDURE:

1. Set up an apparatus similar to Figure 1.
2. Scoop each sample into a plastic cylinder that has a cover of cloth on one end. Cover the cloth end with a cover or cap and tap on the counter 10 times to settle the disturbed sample, remove cover. If the sample arrives in an acetate or metal tube, the hydraulic conductivity is performed on the sample undisturbed. Cut the tubes into appropriate lengths using a saw. Place a cylinder extension on top of the soil tube and seal. Make sure that the seal is watertight. Place a cloth on the bottom of the soil tube.
3. Place samples, cloth-covered end, down into water. Allow the samples to soak at least 16 hours until they are saturated.
4. Turn on the water supply for the upper trough.
5. Transfer the samples to the rack and start the siphons to maintain a constant head of water on each of the samples.
6. After the water level on top of a sample becomes stabilized, collect the percolate in a suitable container for a known amount of time.
7. Record the time and the amount of water in the container. Measure and record the hydraulic head difference (DH). Record the temperature of the water. Measure the length of the soil column and the diameter of the cylinder used.

CALCULATIONS:

$$\text{Hydraulic conductivity} = (Q/A \cdot t) \times (L/\Delta H)$$

Q = Water that passes through the sample at a known time

t = Known time

A = Cross sectional area of the sample

L = Length of the sample

Δ = Hydraulic head difference across sample (See Figure 1)

*Figure 1:



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.34.03

SOP TITLE: Hydraulic Conductivity — Constant Head Method

* Drawing taken from reference listed below

REFERENCES:

Black, C.A. (ed). 1965. Methods of Soil Analysis, Part 1, American Society of Agronomy, Inc., Madison, Wisconsin.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.56.04

SOP TITLE: Texture Procedure — Pipette Method

PURPOSE: To define the analytical procedure used to determine soil texture (% sand, silt, and clay) using a pipette method.

EQUIPMENT:

- Pipettes
- Thermometer
- Stop watch or clock with second's hand
- Balance
- Shaker

REAGENTS:

- 10% Sodium hexametaphosphate Solution: Dissolve 100 GM of sodium hexametaphosphate in 1 L H₂O.

Determine a correction factor by pipetting or dispensing 10 ml of the solution into a pre-weighed container. Place the container in an oven at $105 \pm 5^{\circ}\text{C}$ until dry. Weigh the container on a balance to at least three decimal places. Calculate the correction factor and write it on the container.

$$\text{Blank Factor} = \frac{(\text{Container} + \text{Residue} - \text{Container Empty})}{20}$$

- 30% Hydrogen Peroxide (H₂O₂)
- Acetic Acid

PROCEDURE:

1. Weigh out approximately 10 g of soil. Record the weight on the raw data lab sheet. Weigh in or transfer to a container, such as a fleaker, that has been weighed to at least three decimal places. Follow SOP NUT.01.01 – “Use and Maintenance of Laboratory Balances” to calibrate the balance.
2. **If the % Organic Matter is 1.0 %, Step 2 and 3 are not necessary, skip to step 4.** To the samples that are > 1.0%, add 10 ml of deionized water, 10 ml of 30% Hydrogen Peroxide, and 1 drop of Acetic Acid. Swirl the mixture; cover with a watch glass and heat at approximately 60°C. When the temperature is reached, add Hydrogen Peroxide in 5-ml increments approximately every 15 minutes until the organic matter is oxidized.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.56.04

SOP TITLE: Texture Procedure — Pipette Method

3. After the organic matter is removed, the containers are oven dried at $105 \pm 5^{\circ}\text{C}$, and then weighed to at least three decimal places.
4. Add 10 ml of the 10% Sodium Hexametaphosphate to each container and bring the volume up to approximately 150 ml with deionized water.
5. Cap and shake the containers on a horizontal shaker overnight (12 hours minimum).
6. Add 10 ml of the 10% Sodium Hexametaphosphate solution to a container and bring the volume up to approximately 150-ml. This sample is referred to as a BLANK.
7. The stoppers are then removed and the volume of each container is brought up to 400 ml with deionized water.
8. The temperature of the BLANK is taken and recorded on the raw data lab sheet.
9. Cap the container and shake vigorously for 10 seconds to ensure that no soil adheres to the bottom of the container.
10. Shake the container vigorously for 15 seconds, end-over-end. Note the time and allow the container to stand for the desired settling time based on the temperature of the BLANK (See Table 1).
11. Run the pipette calibration by pipetting DI water into a tarred container and recording the weigh in the appropriate spaces provided on the form. Do this for three consecutive readings. The average is taken for the calibration figure.
12. Pipette 20 ml of the samples, at a depth of 3 cm, at the time corresponding with the temperature of the BLANK (See Table 1). Rinse the pipette by sampling 20 mls of DI water.
13. Each aliquot is discharged into a tarred weighing dish, along with the deionized water used to rinse the pipette.
14. Place the weighing dishes in an oven at $105 \pm 5^{\circ}\text{C}$. When the samples are dry, weigh on a balance to at least three decimal places and record the weight under the “Can Dry Wt.” on the raw data lab sheet. These containers can be dried with the sand containers.
15. Using deionized water, wash the remaining sample through a $45 \mu\text{m}$ sieve.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.56.04

SOP TITLE: Texture Procedure — Pipette Method

16. Transfer the soil from the sieve to a tarred container. Place the sample in a drying oven at $105 \pm 5^\circ\text{C}$ until dry. Weigh the container with the sample on a balance to at least three decimal places and record the weight under the “Sand Dry Wt.” on the raw data lab sheet.

Temperature	Settling Times
$^\circ\text{C}$	hr:min
17	2:37
18	2:33
19	2:29
20	2:26
21	2:22
22	2:18
23	2:16
24	2:13
25	2:10
26	2:7
27	2:4
28	2:1
29	1:58
30	1:56



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.56.04

SOP TITLE: Texture Procedure — Pipette Method

CALCULATIONS:

% Sand = $100 \text{ (Total Sand Weight / Total Soil Weight)}$

% Clay = $(\text{Clay Weight}) (400/\text{Pipet Volume}) (100/\text{Total Soil Weight})$

% Silt = $100 - (\% \text{ Sand} + \% \text{ Clay})$

Where:

Clay Weight = $(\text{Can Dry Wt.} - \text{Can Tare Wt.}) - (\text{Blank Factor})$

Total Sand Weight = $(\text{Sand Dry Wt.}) - (\text{Sand Can Tare Wt.})$

Soil Classification is determined from Textural Triangle

REFERENCES:

Indorante, S.J. March–April 1990. Particle-Size Analysis is a Modified Pipette Procedure. Soil Science Society of America Journal. Volume 54.

Black, C.A. (Ed). 1965. Methods of Soil Analysis, Part 1, American Society of Agronomy, Inc., Madison, Wisconsin.

Isaac, Robert. 1984. Methodology for the Analysis of Soil, Plant, Feed, Water and Fertilizer Samples. Georgia Soil Testing and Plant Analysis Laboratory.

Liegel, E.A., Simson and Schulte (Ed). 1980. Wisconsin Procedures for Soil Testing, Plant Analysis, and Feed and Forage Analysis. Department of Soil Science, University of Wisconsin – Madison.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.58.02

SOP TITLE: Redox Potential of a Soil Sample

PURPOSE: To define the analytical method for determining the oxidation-reduction potential of a soil sample.

EQUIPMENT:

- Redox Electrode
- Millivolt meter
- Thermometer

CALIBRATION:

1. Prepare a Solution of:
105 ml Deionized Water + 3 ml H₂SO₄
2.000 grams of Fe(NH₄)₂(SO₄)₂ * 6H₂O
0.081 grams KMnO₄
Prepare in a beaker and stir with a magnetic stir bar.

Note: It is important that the solution is stirred slowly, to prevent air bubbles.

2. Take a millivolt reading of the above solution when all the solids have dissolved. The millivolt reading should be 474 ± 10 millivolts.

PROCEDURE:

1. Set up small plastic cups in trays.
2. Mark each sample with its appropriate sample number.
3. Scoop the soil samples into the cups using the 15 gram soil scoop, according to SOP NUT.06.02, "Soil Scoop Procedure." Scoop a check sample after the last sample in each tray.
4. Add 15 ml of deionized water to each sample. For each batch of samples, check one aliquot of water with a graduated cylinder and record the volume on the lab sheet.
5. Slowly stir the samples with a glass stir rod.
6. Insert a stir bar into the beaker and record the stabilized millivolt reading while the sample is being stirred slowly.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.58.02

SOP TITLE: Redox Potential of a Soil Sample

7. Record the temperature of the sample.
8. The pH may also be required. Follow the appropriate SOP and record the stabilized reading.

REFERENCE:

Platinum Redox Electrode Instruction Manual. Model 96-78-00, 97-78-00. Orion Research Incorporated, 1991.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.66.03

SOP TITLE: Cation-Exchange Capacity by Sodium Saturation in Calcareous Soils

PURPOSE: To determine the cation exchange capacity of calcareous soil by sodium saturation.

EQUIPMENT:

1. Atomic Absorption Spectrophotometer
2. Vortex mixer
3. Centrifuge

REAGENTS:

SODIUM ACETATE, 1.0N

1. Dissolve 136 g of $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$ in water and dilute to 1000 ml.
2. Adjust the pH to 8.2 with acetic acid or sodium hydroxide solution.

ETHANOL, 95 percent

AMMONIUM ACETATE, 1.0N

(1.0 N Ammonium Acetate Concentrate)

1. Add 400 ml of concentrate to a 2000 ml volumetric flask.
2. Dilute to volume. Mix the solution thoroughly.
3. This makes a 1.0 N Ammonium Acetate solution with a working pH of 7.0.

1.0 N AMMONIUM ACETATE:

	<u>10 L</u>	<u>50 L</u>
Ammonium Hydroxide	690 ml	3450 ml
Acetic Acid	600 ml	3000 ml

1. Rinse out the carboy with deionized water, then fill about 3/4 full.
2. IN (OR IN FRONT OF) THE FUME HOOD use a graduated cylinder to add the Ammonium Hydroxide. Rinse the cylinder with Deionized Water into the carboy.
3. Use the graduated cylinder to add the Acetic Acid. Rinse the cylinder with Deionized Water into the carboy.

DANGER: Ammonium Hydroxide and Acetic Acid both are corrosive and produce corrosive fumes. Use gloves and face protection when handling. Work in (or in front of) the fume hood to avoid inhaling the fumes.

4. Mix the solution by shaking with the lid on or with an electric mixer or a magnetic stir plate and bar.
5. Fill the carboy to volume with deionized water and mix again.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.66.03

SOP TITLE: Cation-Exchange Capacity by Sodium Saturation in Calcareous Soils

6. Check the pH. If the pH is over 7.0, add Acetic Acid. If under 7.0, add Ammonium Hydroxide.

PROCEDURE:

1. Weigh out 4.0 g of soil into a 50 ml conical centrifuge tube.
2. Add 33 ml of sodium acetate solution.
3. Vortex the sample for approximately 10–30 seconds. Shake the sample for 5 minutes.
4. Centrifuge for 5 minutes or until the supernatant is clear.
5. Decant the liquid and discard.
6. Repeat this extraction (Steps 2–4) three more times.
7. Add 33 ml of 95% Ethanol.
8. Vortex the sample for approximately 10–30 seconds. Shake the sample for 5 minutes.
9. Centrifuge for 5 minutes or until the supernatant is clear.
10. Decant the liquid and discard.
11. Repeat this extraction (Steps 7–10) two more times.
12. Add 33 ml of ammonium acetate solution to the sample.
13. Vortex the sample for approximately 10–30 seconds. Shake the sample for 5 minutes.
14. Centrifuge for 5 minutes or until the supernatant is clear.
15. Decant the liquid into a 100 ml volumetric flask.
16. Repeat this extraction (Steps 12–15) two more times.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.66.03

SOP TITLE: Cation-Exchange Capacity by Sodium Saturation in Calcareous Soils

17. Dilute the ammonium acetate extract to volume and mix.
18. Determine the sodium by using the method and standards in SOP NUT.02.12. – “Procedures for the Determination of Ammonium Acetate Extractable Calcium, Magnesium, Sodium and Potassium in Soil.”
19. If the samples exceed the highest standard by more than 10 %, an appropriate dilution is made and calculated.

CALCULATION:

Cation Exchange Capacity by Sodium Saturation (meq/100g) =
Atomic Absorption Reading(ppm) * .01087

REFERENCES:

Homer D. Chapman and Parker F. Pratt. 1978. Methods of Analysis for Soil, Plants, and Waters. Division of Agricultural Sciences. pp. 35–36.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.92.00

SOP TITLE: Determination of Total Organic Carbon in Soil by Combustion

EQUIPMENT:

- LECO CR-412 Carbon Analyzer
- Weigh Boats

PROCEDURE:

1. Instrument Set-Up:

- A. Check the O₂ supply to furnace and set regulator to 40 psi as stated in the manual.
- B. Power up the furnace and allow it to come to temperature, about 1400°C.
- C. Check for proper gas flow as stated in the user manual for this system.
- D. Replace Anhydrone™ if instrument indicates it is wet.
- E. Check the Ambient Monitor for proper instrument conditions. Print table and place it in the appropriate calibration logbook. All data must be signed and dated by the individual generating the data.

2. Calibration:

- A. Calibration data is stored in the calibration logbook.
 - B. Check current calibration curve by combusting three replicates (approximately 0.2g of a known low standard) as detailed in the system calibration logbook. Document information on the standard such as supplier, lot number, and ppm concentration in the calibration logbook.
3. System should be recalibrated if:
1. The value of standard varies more than 5% relative to the expected value.
 2. Carbon content of unknown samples is expected to be above the current calibration curve.
 3. The oxygen tank is changed.
 4. The CR-412 Carbon Analyzer has sat idle for four weeks or more.
- D. Calibrate by combusting at least 7 replicates of the known low and high ppm standard, 0.2g within 5% and 0.09g within 5%, respectively.
 - E. Record calibration data in the appropriate calibration logbook. All data must be signed and dated by the individual generating the data.

3. Sample Analysis:

- A. All samples tested for organic carbon content must be checked for inorganic carbon by placing several drops of 2M HCl on a small amount of soil (approximately 0.2g) to check for carbonates. If no carbonates are present (no effervescence observed), place approximately 0.25g into a weighing dish and heat at 104°C (2 to 16 hours) to allow carbon to be determined on an oven dry weight basis. Store samples in a desiccator until ready to weigh.



STANDARD OPERATING PROCEDURE

SOP NUMBER: NUT.02.92.00

SOP TITLE: Determination of Total Organic Carbon in Soil by Combustion

- B. Follow this procedure if carbonates are present as indicated by effervescence. Place enough soil in a weighing dish for the desired reps allowing 0.25g/rep. Next, add 2M HCl until effervescence stops. Oven dry the samples as in Step A for noncalcareous soils.
- C. Weigh about 0.200g of the oven dried soil into a combustion crucible and record the weight. Manually enter the recorded weight in the instruments analysis screen menu.
- D. Percent carbon is calculated by the instrument and printed. Record this value on the appropriate lab sheet.

4. System Shutdown:

- A. Shutdown the system by choosing “**LOG OFF**” on the screen. The furnace temperature will be reduced and oxygen flow through the instrument will be stopped.

REFERENCES:

LECO CR-412 Instrument manual, revised 1995.



STANDARD OPERATING PROCEDURE

SOP TITLE: 1/3 Bar — German Method

Subsequently, the following operations are carried out:

- 1) Taring of Buchner funnels with premoistened filter (A, A', A'')

MWC¹-determination:

- 2) One aliquotes of approx. 100 g each (filled to the rim) are divided into tared Buchner funnels. Soil allowed again to drip off. Soil is saturated and allowed to stand overnight. Funnels to be covered with a reversed watch glass in order to prevent drying out of the soil.

FC²-determination: 1/3 Bar

- 4) Covering/sealing of Buchner funnels with 1–2 layers of parafilm and slight pressing down of film on soil. Tightness of parafilm at the Buchner funnel to be checked so that no air current can go through the soil. This step serves to prepare for the FC-determination.

- 5) Application of a saturation potential of 1/3 bar to the suction bottles (vacuum regulator setting: –0.33 bar, manometer setting: 666 mbar),

- 0.33 bar should not fall below otherwise high pressure is exerted on the soil via the parafilm membrane and, thus, too much water is pressed out. Increase vacuum only slowly from 0 to –0.33 bar.

With this procedure the water starts to emerge. Suction to be used until dripping off speed is only approx. 1 drop/15 minutes (at least, however, 2 hours, attention to be paid to tightness of the parafilm).

- 6) Balance of the low pressure to be established and parafilm removed.
- 7) The funnels with the partially wet soils to be weighed.
- 8) Soil is dried in the Buchner funnels.

¹ The MWC is frequently described in American literature as FC (field capacity, pF ≤ 0.3)

² The FC is frequently described in American literature as 1/3 bar-FMC (field moisture capacity, pF ≤ 2.5)

